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Ion irradiation effect (Ar^+ , $E = 20\text{--}40$ keV) on the mechanical properties and microstructure of aluminum V95 alloy

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Abstract. The effect of 20–40 keV Ar^+ ion irradiation on the mechanical properties and structural and phase state of hot-pressed profiles 6 mm thick made of an V95 alloy (Al–Zn–Mg–Cu system) after artificial aging has been studied. Irradiation (without causing heating) at fluences of $2 \cdot 10^{15} \text{ cm}^{-2}$ and $1 \cdot 10^{16} \text{ cm}^{-2}$ has been revealed to increase the relative elongation by 5%, without changing the strength characteristics. Electron microscopy examination has showed that irradiation has an effect on the state of the subgrain structure of the alloy. Irradiation at these fluences results in partial enlargement of the subgrain structure, and at higher fluence of $9.4 \cdot 10^{16} \text{ cm}^{-2}$ it completely transforms the structure into coarse-grained one. At a distance of 150 μm from the irradiated surface, there is a slight increase in the subgrain size. The irradiation changes the morphology of $\text{Al}_6(\text{Fe}, \text{Mn})$ intermetallic compounds in both the surface layer and the sample volume; namely, it sharply decreases the bulk density of lath-shaped intermetallic compounds and increases the density of the equiaxed ones. The degree of influence depend on the irradiation mode.

1. Introduction

This work is aimed at studying the mechanical properties, structure, and phase composition of V95 alloy samples cut from 6-mm-thick hot-pressed profiles in their initial state after artificial aging ($T = 140^\circ\text{C}$, 16 h) and after their subsequent Ar^+ ion irradiation at ion energies of 20–40 keV.

Alloy V95 is one of the most universal and widespread construction materials of the Al–Zn–Mg–Cu system. It is commonly used to manufacture parts that operate under high loads.

The artificially-aged V95 alloy has a high corrosion resistance. Among all aluminum alloys, the V95 alloy has the maximum strength, but its plastic properties are low. Therefore, it is of interest to search for opportunities to enhance the microstructure and plastic properties of the alloy using ion-beam treatment, which has been successfully proven in recent years as a technique for a specific modification of the properties of functional materials.

The application of ion-beam treatment is reasonable due to the fact that the exposure depth during



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single-sided irradiation can reach several millimeters when the ranges of low and medium energy ions ($E < 10^6$ eV) in the substance are only a part of micron. This is due to nanoscale post-cascade radiation-dynamic effects during ion bombardment, which are experimentally discovered and theoretically validated in the papers [1–3].

2. Experimental

The V95 alloy profiles after quenching and artificial aging (140 °C, 16 h) were irradiated with continuous Ar^+ ion beams in an ILM-1 ion beam implanter equipped with a PULSAR-1M ion source based on a glow discharge with a hollow cold cathode [4]. The profiles were cut into samples 20 cm long. The samples during irradiation were suspended (by the corners) to avoid their uneven heating. A line-focus beam 100×20 mm² in cross section was cut out from the ion beam of circular cross section using a collimator. The samples exposed to the ion beam were moved at a speed of 2 cm/s. The temperature of the targets was continuously controlled with the help of a chromel-alumel thermocouple, which was metrologically tested and welded to an identical test sample. We used the following irradiation modes: (1) $E = 20$ keV, $j = 400$ $\mu\text{A}/\text{cm}^2$, $F = 2 \cdot 10^{15}$ cm⁻²; (2) $E = 20$ keV, $j = 400$ $\mu\text{A}/\text{cm}^2$, $F = 1 \cdot 10^{16}$ cm⁻²; and (3) $E = 40$ keV, $j = 500$ $\mu\text{A}/\text{cm}^2$, $F = 9.4 \cdot 10^{16}$ cm⁻². The maximum temperature to which the samples were heated during irradiation was in the range from 30 to 200 °C.

The static tests for uniaxial tension were conducted at room temperature, according to the standard technique (State Standard GOST 1497-84). The measurement error was ~5 %. Electron microscopy analysis was carried out by the method of thin foils using a JEM-200 CX transmission electron microscope, at the Electron Microscopy Center of Collaborative Access of the Institute of Metal Physics, UB RAS. The microstructure was examined in two cross-sections parallel to the irradiated surface: (1) directly near the surface and (2) at a distance of ~150 μm from it.

3. Details of exposure, measurements and results

Table 1 presents mechanical properties of the samples cut from the extruded V95 alloy profiles, which were measured in the initial state and after irradiation.

Table 1. Mechanical properties of the V95 alloy samples in the initial state (after aging) and after various modes of Ar^+ ion beam irradiation.

No	Alloy processing	$\sigma_{0.2}$, MPa	σ_u , MPa	δ , %
1	Initial state after quenching and artificial aging (140 °C, 16 h)	496.8	605.8	10.0
2	Irradiation mode 1: $E = 20$ keV, $j = 400$ $\mu\text{A}/\text{cm}^2$, $F = 2 \cdot 10^{15}$ cm ⁻² , $T = 30$ °C	515.1	631.6	15.4
3	Irradiation mode 2: $E = 20$ keV, $j = 400$ $\mu\text{A}/\text{cm}^2$, $F = 1 \cdot 10^{16}$ cm ⁻² , $T = 40$ °C	541.4	595.6	15.4
4	Irradiation mode 3: $E = 40$ keV, $j = 500$ $\mu\text{A}/\text{cm}^2$, $F = 9.4 \cdot 10^{16}$ cm ⁻² , $T = 200$ °C	362.4	450.5	17.8

Table 1 suggests that the ion beam treatment of the alloy under used irradiation conditions (without heating the samples to significant temperatures, $T \sim 30$ – 40 °C) does not change the ultimate strength and yield strength within the measuring error; however, it increases the relative elongation by 5 %. Irradiation in the mode 3 at higher ion energy and ion current density (causing the ion-beam-induced heating of the samples to 200 °C) decreases the strength properties significantly. The mechanical properties acquired in this mode do not meet the regulated requirements.

Electron-microscopic examination of the V95 alloy in its initial state indicates a developed subgrain structure (figure 1 a). Subgrains have an equiaxed shape and an average diameter of 1–5 μm . There are lath-shaped intermetallic $\text{Al}_6(\text{Fe}, \text{Mn})$ compounds 0.2–0.5 μm long of the crystallization origin in the volume of subgrains (figure 1 a, b). The subgrains exhibit a contrast from the fine

hardening phases precipitated during artificial aging. So, $\theta'(\theta'')$ particles (metastable modifications of the stable θ phase of CuAl_2) are observed in the form of flat discs ~ 20 nm in diameter precipitated along the $\{001\}\text{Al}$ planes (figure 1 *c*), as well as η' and η (MgZn_2) phase particles 5–10 nm in diameter, which are uniformly distributed over the $\{111\}\text{Al}$ planes (figure 1 *d*). Using the method of analysis carried out in [5], it was found that the alloy mainly contains precipitates of the coherent θ'' and η' phases.

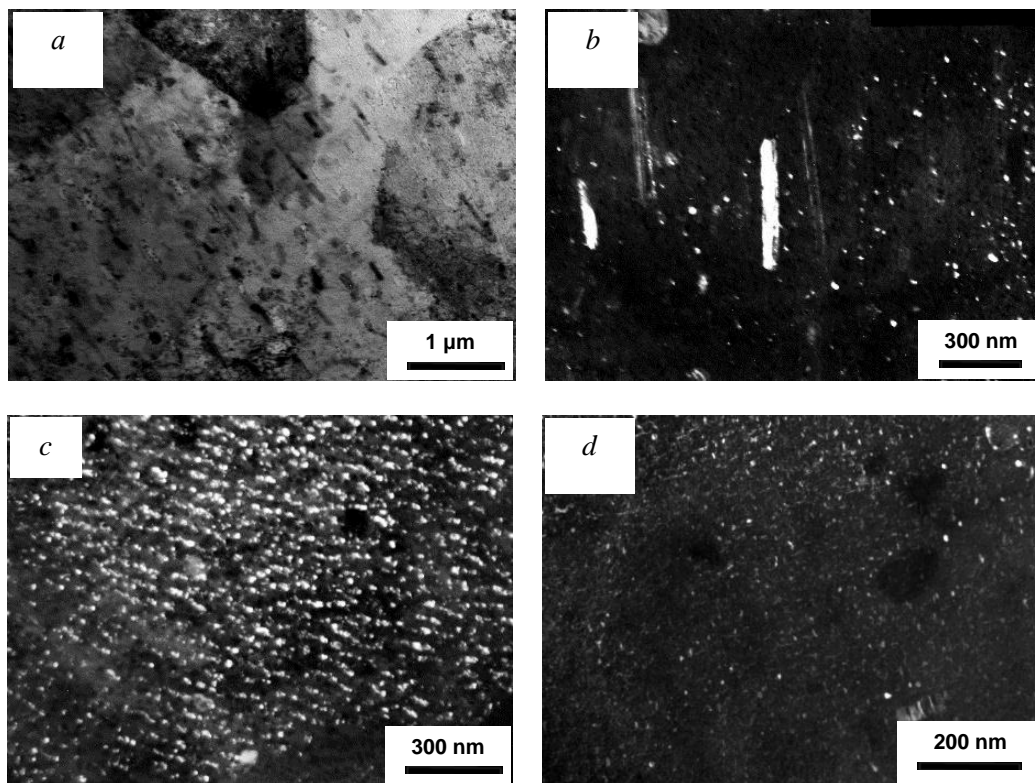


Figure 1. Microstructure of the V95 alloy after quenching and artificial aging (140 °C, 16 h): (a) bright-field image and (b–d) dark-field images taken in the following reflections: (b) $(222)\text{Al}_6(\text{Fe},\text{Mn})$, (c) $(200)\theta''(\theta')$, and (d) $(203)\eta' + (104)\eta$.

Irradiation in mode 1 ($E = 20$ keV, $j = 400 \mu\text{A}/\text{cm}^2$, $F = 2 \cdot 10^{15} \text{ cm}^{-2}$) leads to the formation of a mixed grain-subgrain structure in the surface of the sample: There are regions with coarse-grained structure where the size of some crystals (grains) exceeds $10 \mu\text{m}$ (its fragment is shown in figure 2 *a*), and regions with subgrained structure where the diameter of some subgrains is $2\text{--}5 \mu\text{m}$ (figure 2 *b*). There are dense dislocation tangles in the grains and subgrains. At a distance from the irradiated surface, mainly equiaxed subgrains with a diameter of $2\text{--}5 \mu\text{m}$ are detected. After irradiation in mode 1, both near the surface and at a depth of $\sim 150 \mu\text{m}$ from it, lath-shaped $\text{Al}_6(\text{Fe},\text{Mn})$ intermetallics to $0.3 \mu\text{m}$ long remain in the alloy (figure 2 *a*). The density of their distribution as a result of irradiation decreases. In addition, equiaxed $\text{Al}_6(\text{Fe},\text{Mn})$ particles $50\text{--}80$ nm in size form (figure 2 *c*). There are hardening η' and η phases in the irradiated alloy as well as in the aged alloy (figure 2 *d*). The diameter of the η' -phase particles reaches 5 nm and that of η -phase particles, 10 nm. The total density of their distribution is high, while stable phase precipitations predominate. No copper-bearing $\theta'(\theta'')$ phases were detected in the irradiated sample. Most likely, this is due to an increase in the volume fraction of η - and η' -phase [6].

A similar structural-phase state was revealed in the V95 samples after irradiation in mode 2 at the same ion energy and ion current density, but a higher fluence (see table 1).

Irradiation in mode 3 ($E = 40$ keV, $j = 500 \mu\text{A}/\text{cm}^2$, $F = 9.4 \cdot 10^{16} \text{ cm}^{-2}$), which is accompanied by

sample heating to 200 °C, results in the formation of a coarse-grained structure near the irradiated sample surface. In the volume, at a depth of ~ 150 μm , the developed subgrain structure observed in the initial state remains. The size of subgrains increases slightly under irradiation (approximately to 4–8 μm). There are $\text{Al}_6(\text{Fe,Mn})$ intermetallics in the form of laths 0.2–0.4 μm long and equiaxed particles ~ 0.1 μm in diameter found both in the sample volume and near the irradiated surface. After irradiation in the considered mode, magnesium-zinc η and η' , as well as aluminum-copper θ'' and θ' hardening phases are also present in the studied alloys. Moreover, a decrease in the distribution density of the stable phase η (diameter ~ 10 nm) and an increase in the low-contrast metastable phase η' (diameter less than 5 nm) are observed. A decrease in the amount of the stable η phase, according to [6], stimulates the precipitation of the aluminum-copper θ'' (θ') phase. A similar trend was observed for the initial artificially aged state (since copper atoms partially dissolve in the stable η phase). Contrast analysis of the particles in the images indicates the coexistence of disc-shaped coherent θ'' -phase precipitates with a diameter to 20 nm and partially coherent θ' precipitates of the same shape with a diameter of 50–70 nm.

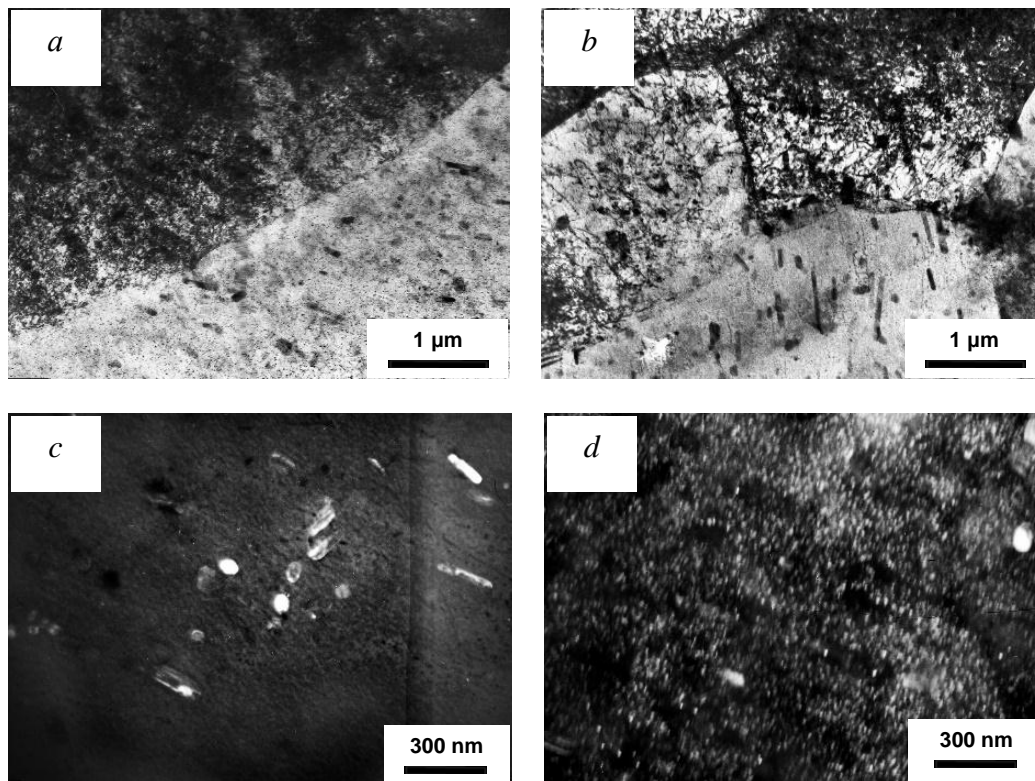


Figure 2. Microstructure of the V95 alloy after irradiation: $E = 20$ keV, $j = 400$ $\mu\text{A}/\text{cm}^2$, $F = 2 \cdot 10^{15}$ cm^{-2} : (a), (b) bright-field and (c), (d) dark-field images taken in the (c) $(222)\text{Al}_6(\text{Fe,Mn})$ and (d) $(200)\eta' + (004)\eta$ reflections.

The results of the electron-microscopic examination suggest that the irradiation accompanied by sample heating induces the dissolution of the hardening η' and η (MgZn_2), θ'' (θ') (CuAl_2) phases existing in the initial state and the formation of a supersaturated α -solid solution. This occurs both near the irradiated surface and in the sample volume. The α solid solution decomposes during subsequent storage due to natural aging of the irradiated sample and these phases precipitate again. The metastable η' phase prevails in the naturally aged alloy. The small amount of stable η phase and a high density of point defects such as irradiation-induced vacancies initiate the precipitation of θ'' -phases and partial of $\theta'' \rightarrow \theta'$ transformation.

4. Conclusions

This work showed that 20-keV Ar⁺ ion irradiation had an influence on the mechanical properties and the structural-phase state of the 6-mm-thick hot-pressed and artificially-aged V95 alloy profiles. Irradiation with 20-keV Ar⁺ ions ($j = 400 \mu\text{A}/\text{cm}^2$) at fluences of $2 \cdot 10^{15}$ and $1 \cdot 10^{16} \text{ cm}^{-2}$ (that did not cause heating) increased the relative elongation of the samples by 5 %, while the strength characteristics remained the same. The increase in the ion energy to 40 keV, ion current density to $500 \mu\text{A}/\text{cm}^2$, fluence to $9.4 \cdot 10^{16} \text{ cm}^{-2}$ and heating to 200 °C deteriorated the mechanical properties of the samples significantly.

The irradiation effect on the initial subgrain structure was revealed to decrease with increasing distance from the irradiated surface. The subgrain structure near this surface becomes either significantly enlarged or partially or completely transformed into coarse-grained one, depending on the irradiation mode. However, there is only a slight increase of subgrain sizes in all cases in the sample volume. The irradiation changes the morphology of Al₆(Fe,Mn) intermetallic compounds; namely, the volume density of lath intermetallic compounds decreases, whereas the volume density of equiaxed intermetallic compounds sharply increases. The solid solution decomposition process also depends on the irradiation mode. Irradiation at fluences of $2 \cdot 10^{15}$ and $1 \cdot 10^{16} \text{ cm}^{-2}$ initiates the $\eta' \rightarrow \eta$ (MgZn₂) transformation and suppresses the nucleation and growth of the metastable aluminum-copper θ'' (θ') phases (CuAl₂). Irradiation at fluences of $9.4 \cdot 10^{16} \text{ cm}^{-2}$, on the contrary, reduces the amount of the stable η phase and the number of θ'' - and θ' -phase particles, which appear to form during natural aging of supersaturated solid solution during subsequent storage of the irradiated sample.

The result achieved after low dose irradiation without heating the samples can improve life properties.

Acknowledgments

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